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The Influence of Various Variables on In Situ Transesterification of *J. curcas*

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Process Intensification Group

Bionergy III, May 22 - 27, 2011

Methyl Ester

- METHYL ESTER (BIODIESEL)
  - Renewable fuel for diesel engines derived from natural oils which meet the specification (ASTM D 6751, EN 14214)
  - Composed of fatty acids alkyl ester
  - Transported and sold using existing infrastructure

Methyl Ester

- 4 factors determining biodiesel competitiveness as fuel
  - Feedstock price
  - Biodiesel production cost
  - Policy (tax, incentive) on biodiesel
  - Crude oil prices

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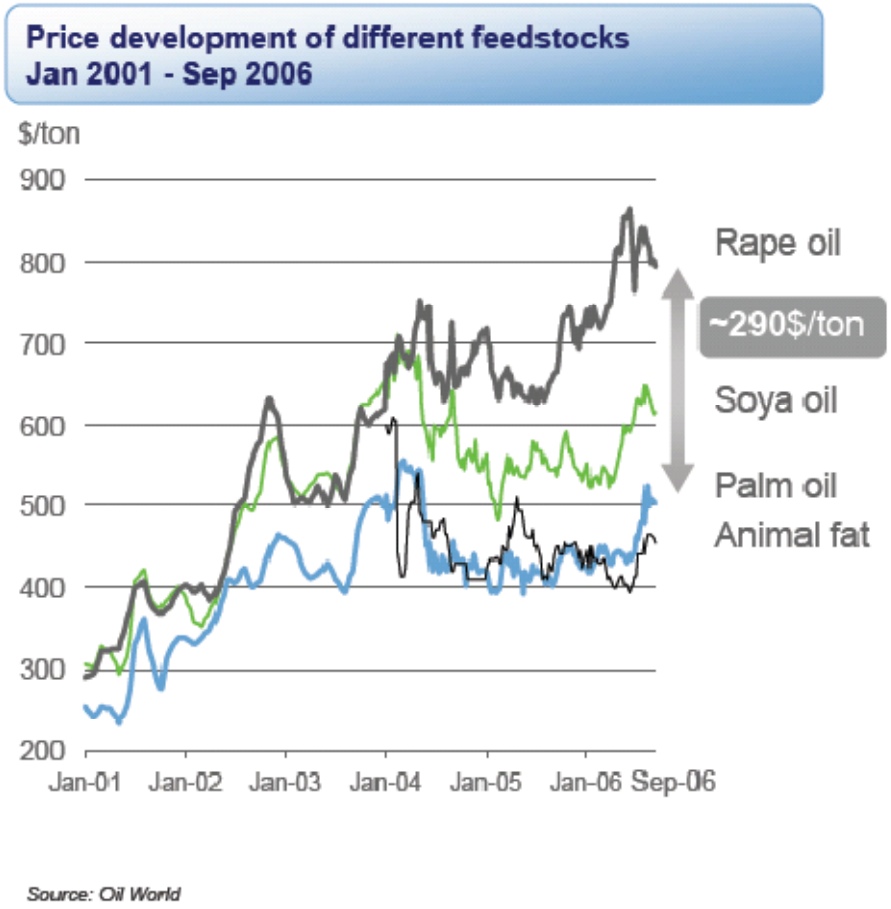
Feedstock Price

(Edible oil)



Source: Kimmo Rahkamo, Neste Oil presentation material

Feedstock Price

Fatty acid composition in various non edible oils (Azam *et al.*, 2005)

Fatty acid		*J.curcas*	*P. pinnata*	*S. oleidis*	*A. indica*
Capric	10:0			0.8	
Lauric	12:0			35.6	
Myristic	14:0	1.4		50.7	
Palmitic	16:0	15.6	10.6	4.5	14.9
Stearic	18:0	9.7	6.8		14.4
Oleic	18:1	40.8	49.4	8.3	61.9
Linoleic	18:2	32.1	19	0.1	7.5
Arachidic	20:0	0.4	4.1		1.3
Eicosenoic	20:1		2.4		
Behinic	22:0		5.3		
Lignoceric	24:0		2.4		

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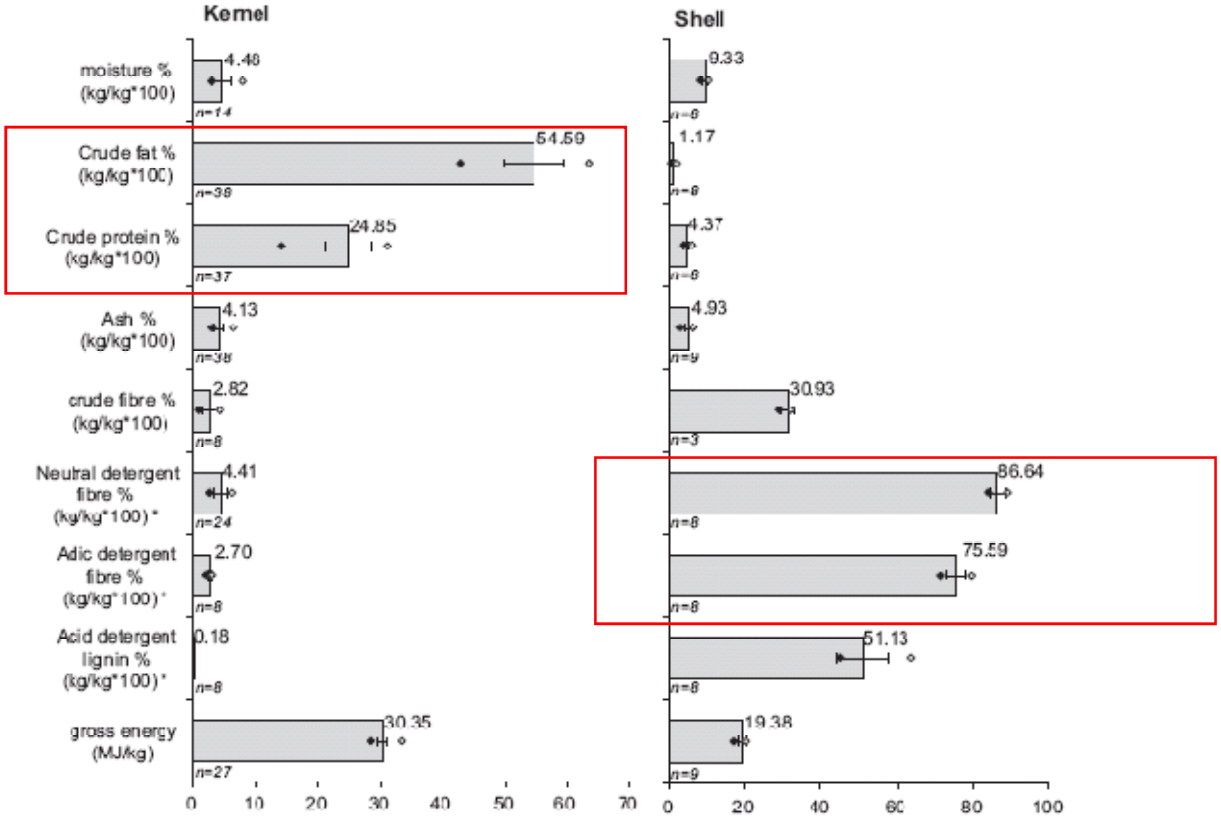
•The kernel contains mostly fat and protein while the shell dominates by fiber

• Oil content varies very much, but generally between 30 – 60%



- Problems (1):
  - Via conventional TE, when alkali catalyst is employed, high FFA contents in the JC oil promotes saponification rather than TE reaction and produce soap rather than methyl ester

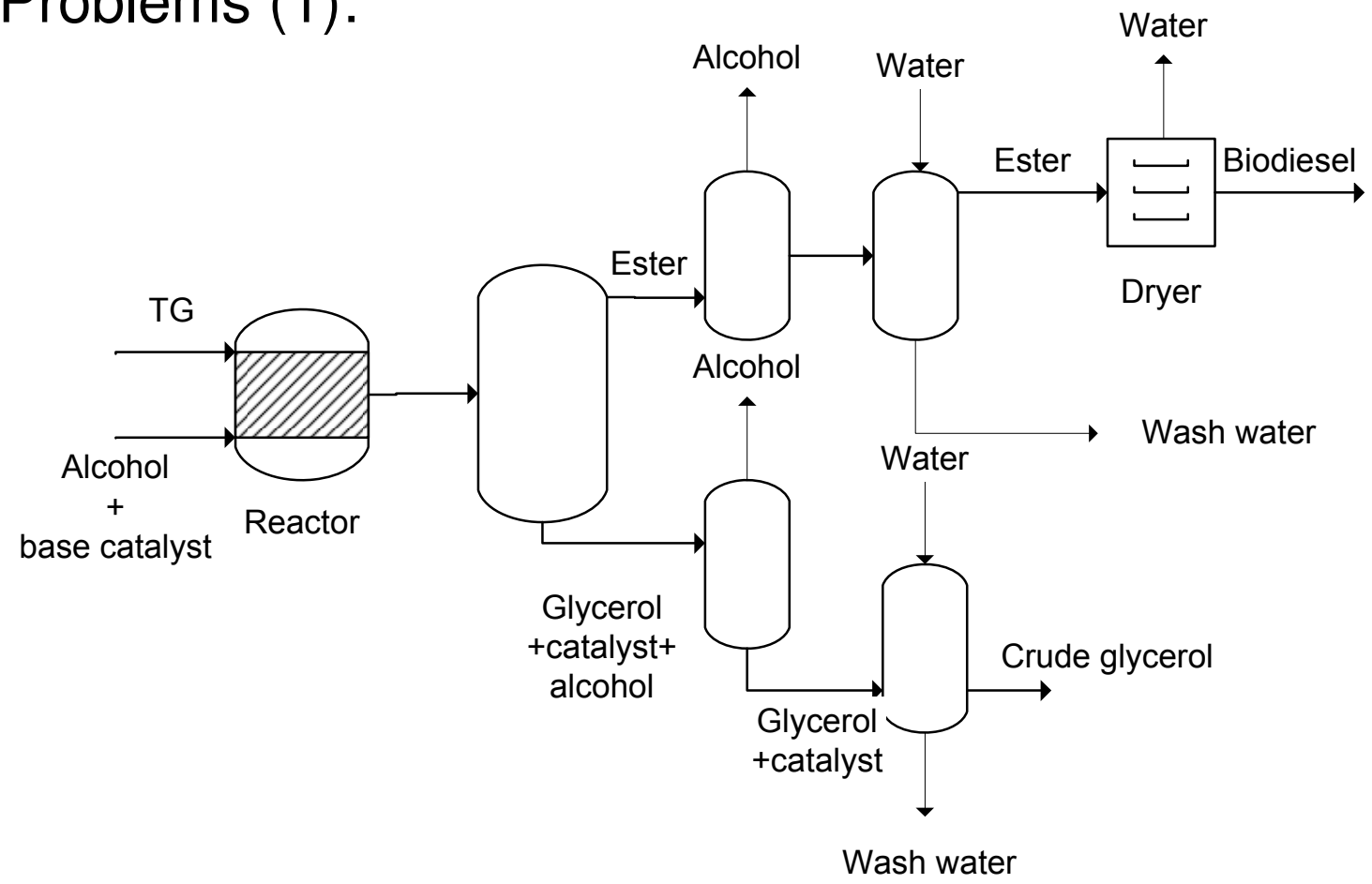
$$RCOOH + NaOH \leftrightarrow RCOONa + H_2O$$

FFA Soap Water

  - Consume the catalyst and halt the TE process. Effect on yield as well as on downstream processing

Conventional T.E

- Problems (1):



Conventional TE with high FFA raw material

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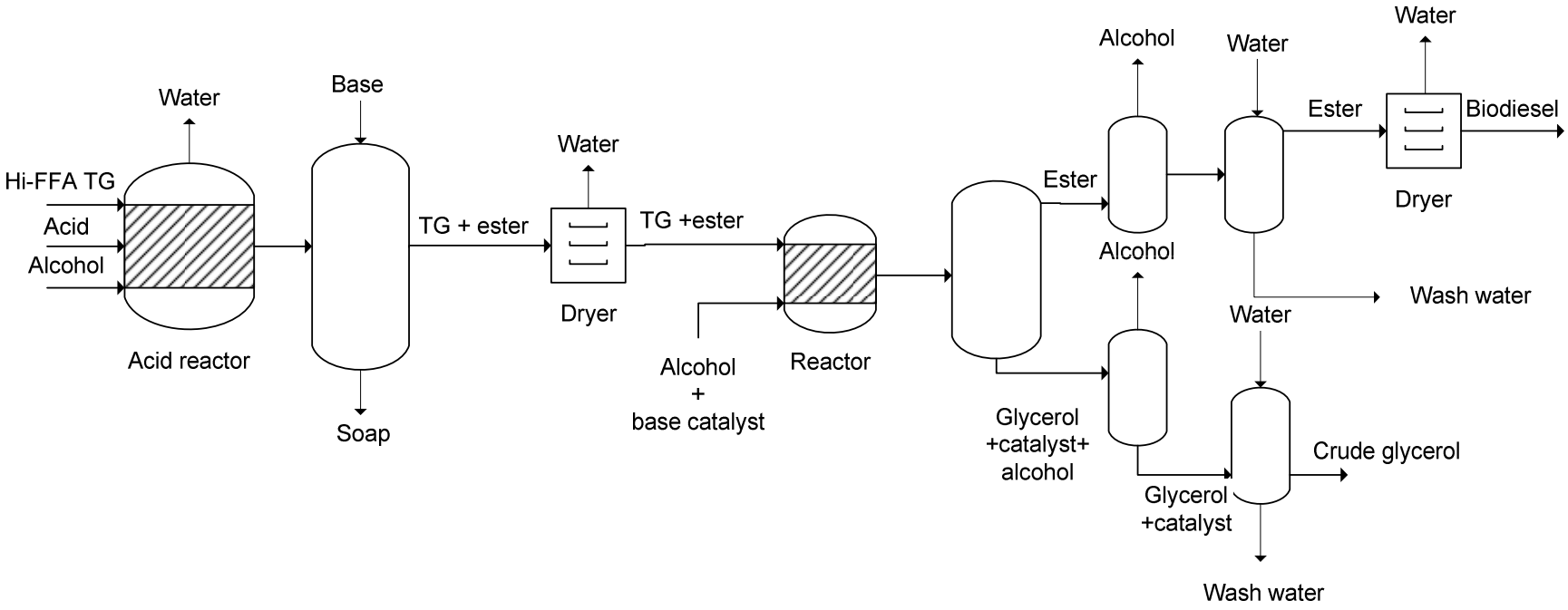

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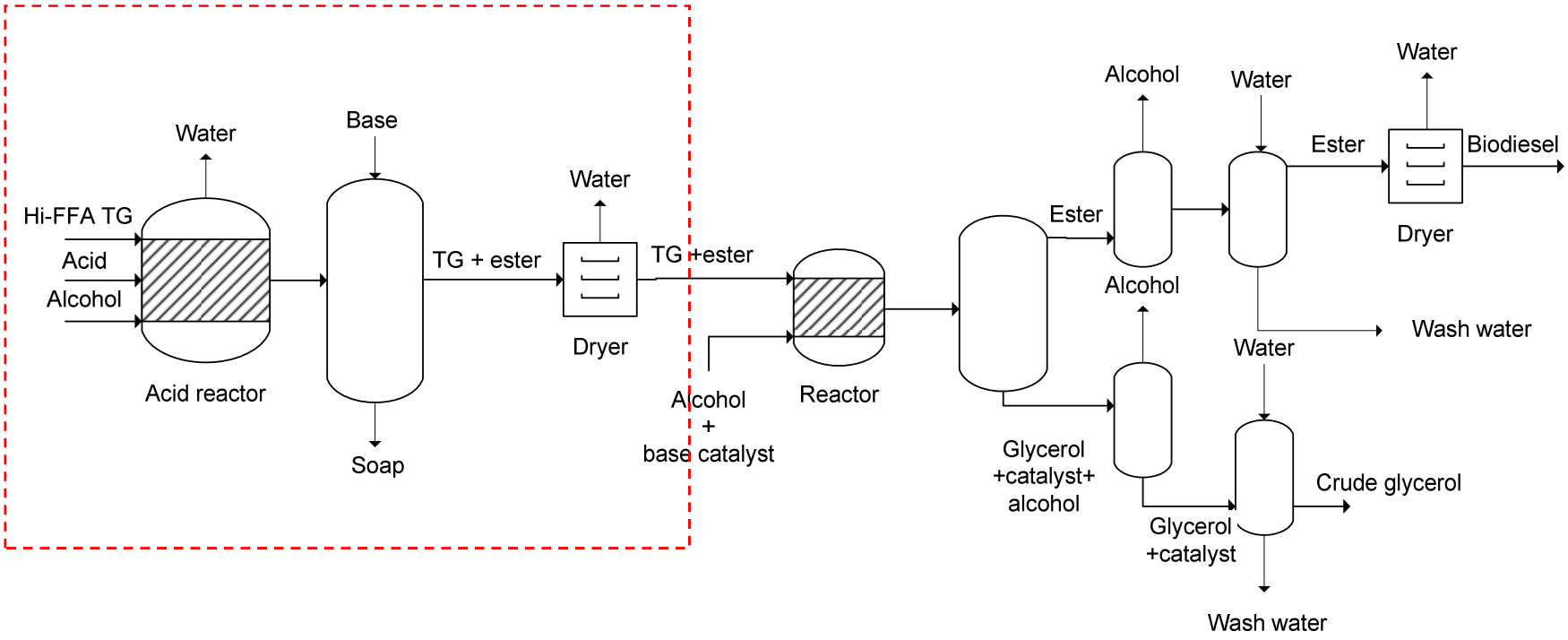
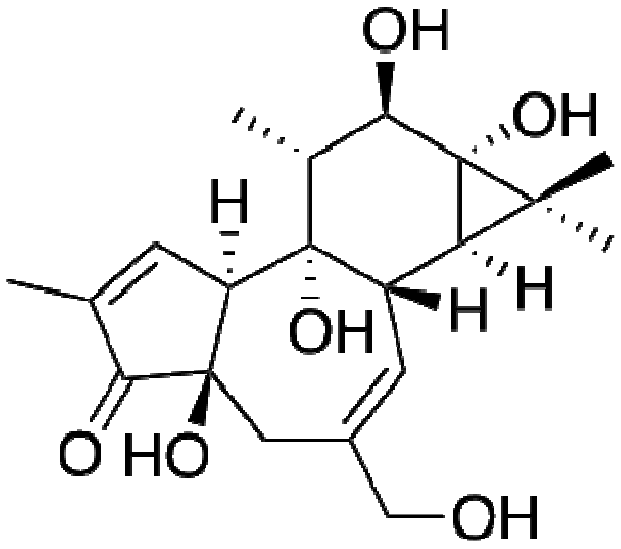
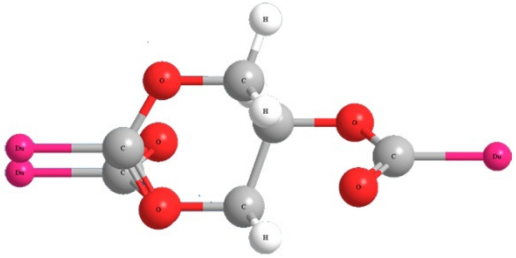


PROBLEM 2

- Phorbol esters in JC are toxic, even in low concentration
- The phorbol esters end up in the meal, make the meal unsuitable for animal feed
- There is a report that phorbol esters decompose in access methanol, but there is no report on the fate of phorbol esters in in situ transesterification of JC

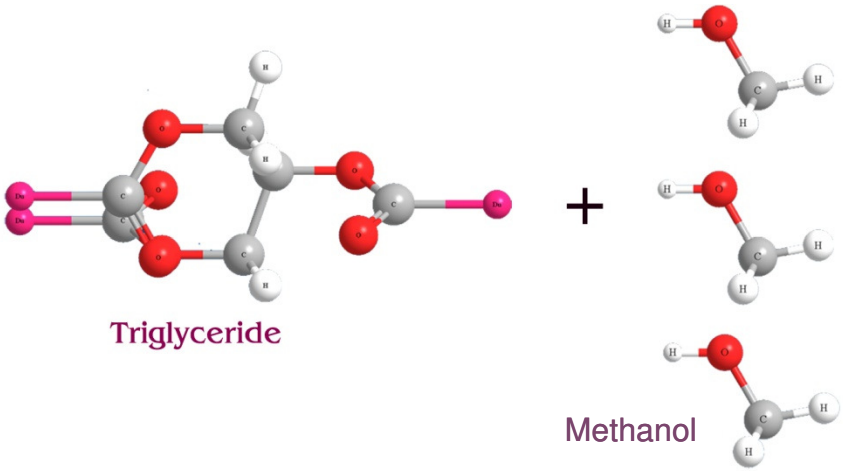


Biodiesel Production



Triglyceride

Biodiesel Production



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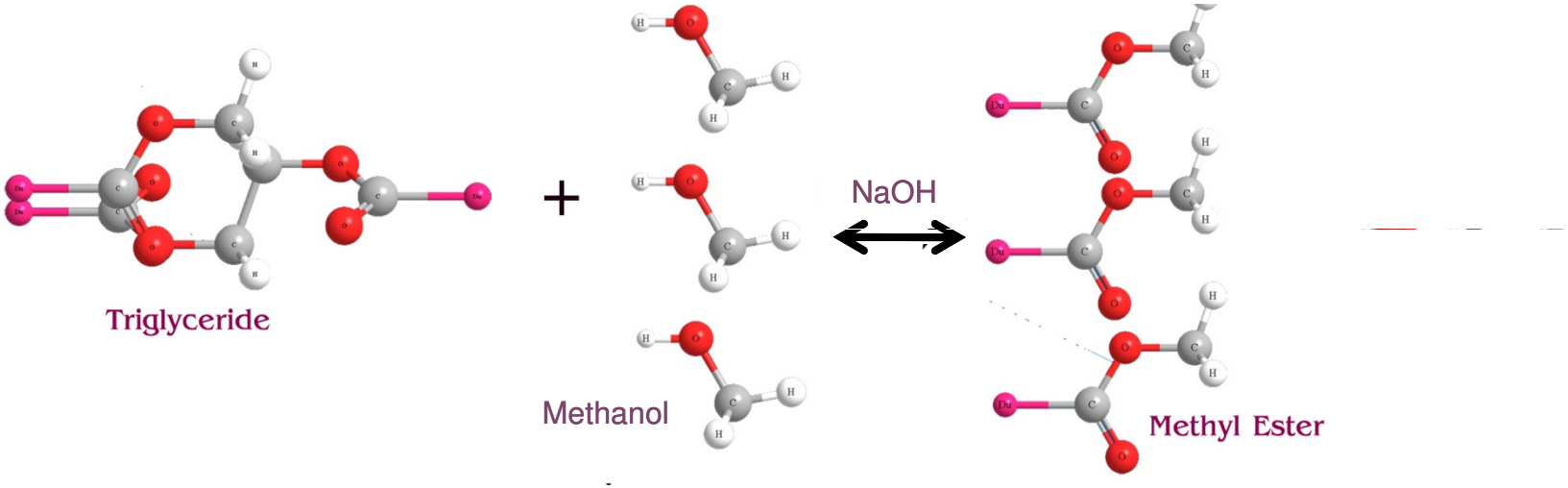

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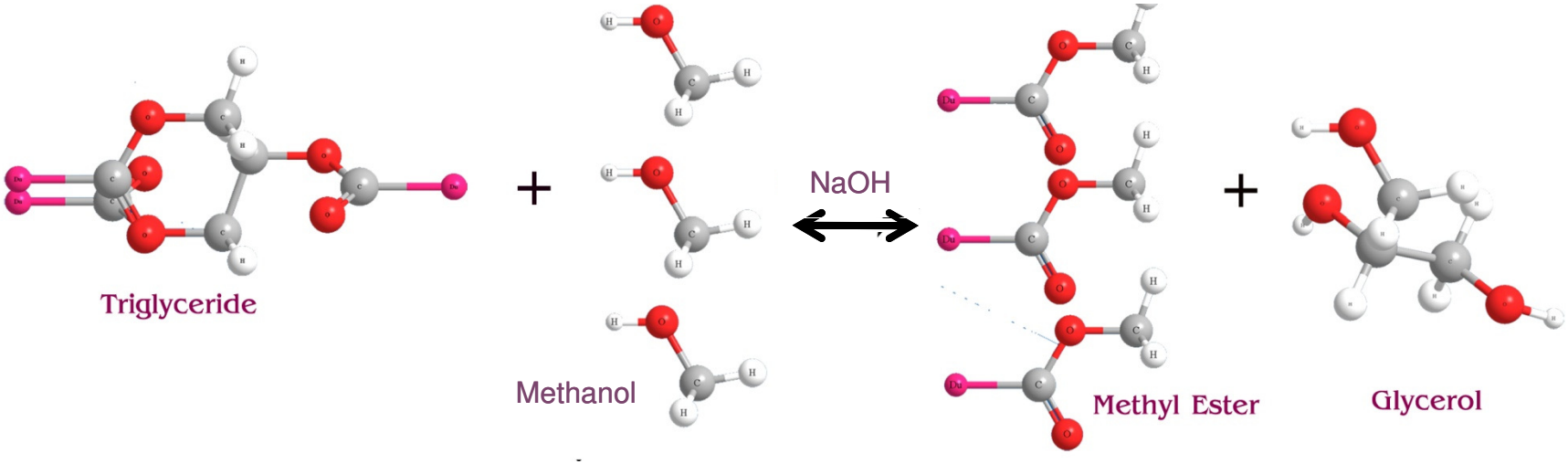
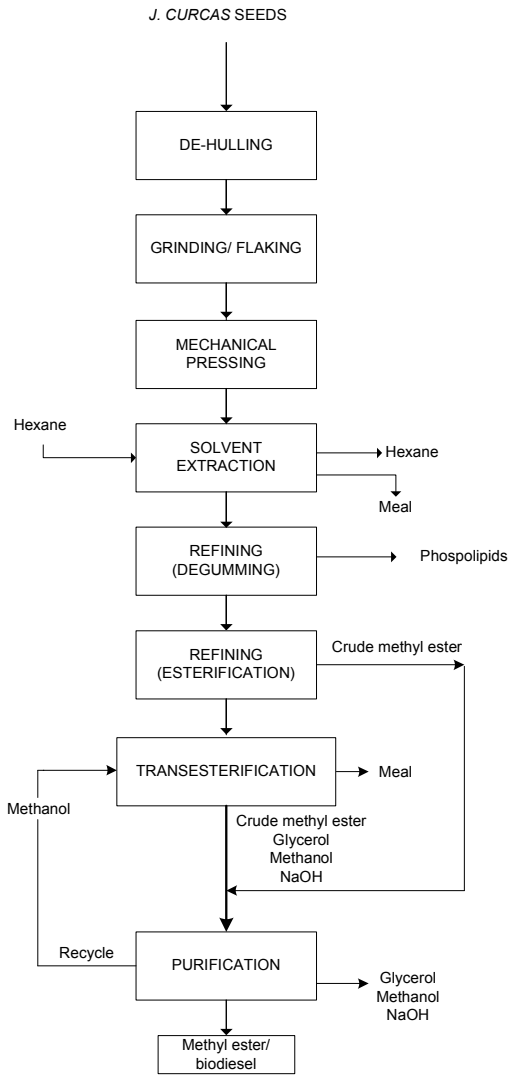
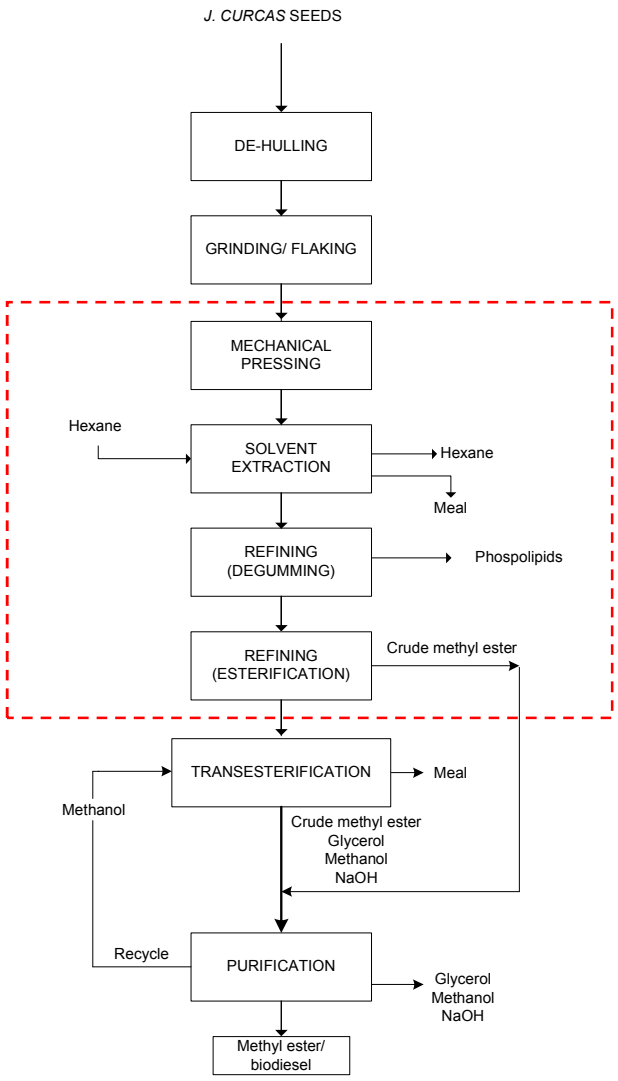




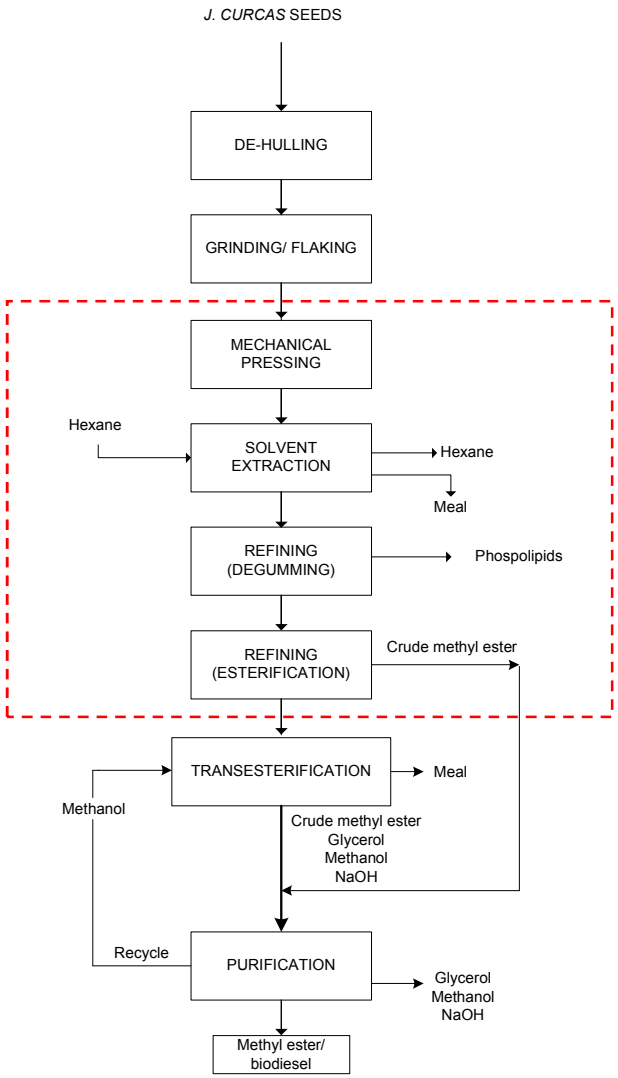
Conventional biodesel process from *J.curcas* seeds

- Conventional transesterification of oil-bearing materials.



Conventional biodesel process from *J.curcas* seeds

- In situ transesterification is the direct transesterification of ground oil-bearing materials.



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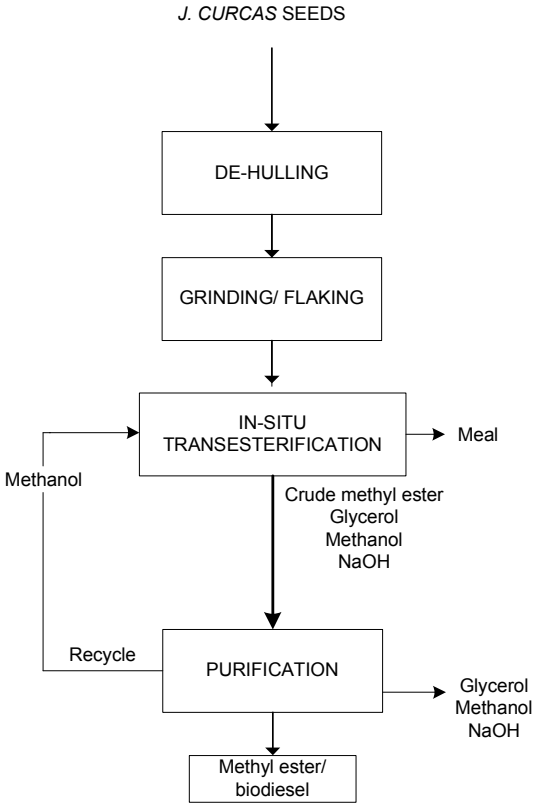
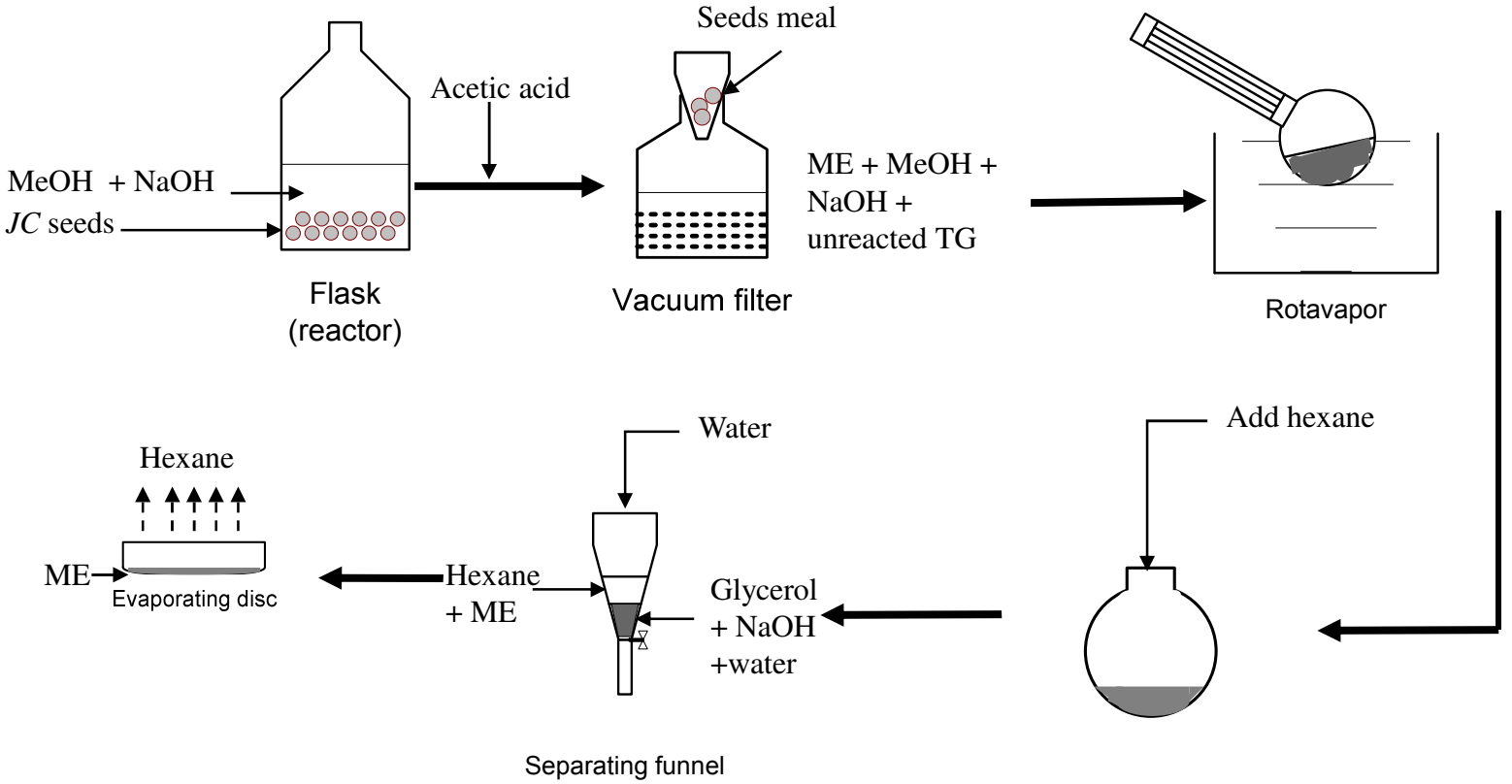


In-situ transesterification/ reactive extraction biodesel process from *J.curcas* seeds


MeOH + NaOH
JC seeds
Flask
(reactor)
Acetic acid
Seeds meal
Vacuum filter
ME + MeOH +
NaOH +
unreacted TG
Rotavapor
Add hexane
Water
Glycerol
+ NaOH
+water
Separating funnel
Hexane
+ ME
Hexane
ME
Evaporating disc

Factor	Unit	Code	Level	
			-1	+1
Molar ratio of methanol to oil	-	A	100	400
NaOH concentration	N	B	0.1	0.2
Reaction time	Min	C	10	60
Reaction temperature	^{o}C	D	30	60
Mixing speed	rpm	E	100	400

Two level factorial design

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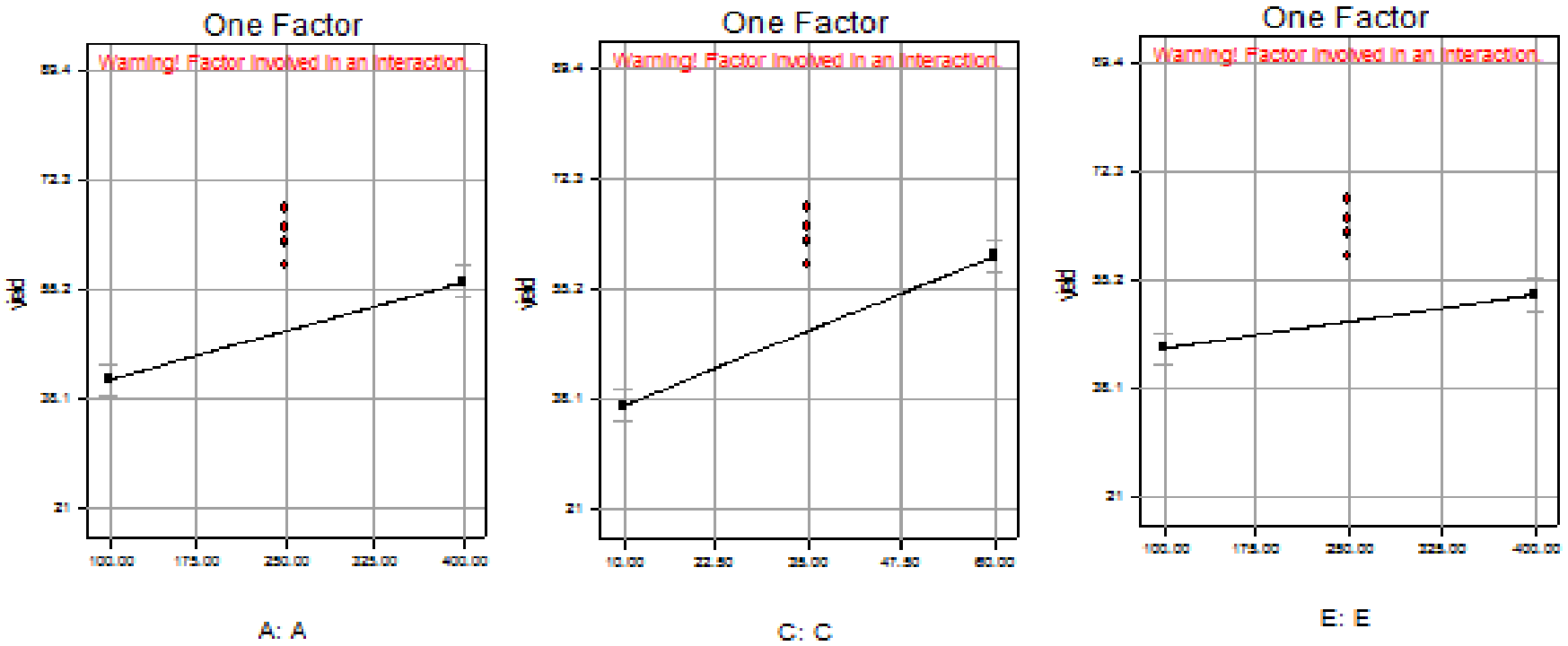
- Executed on Design Expert software
- Model used was a 2^5 full factorial design
- Model based on first order model
- 5 central points were added to the design to assess the experimental error and also the curvature
- Overall there were 37 experiments



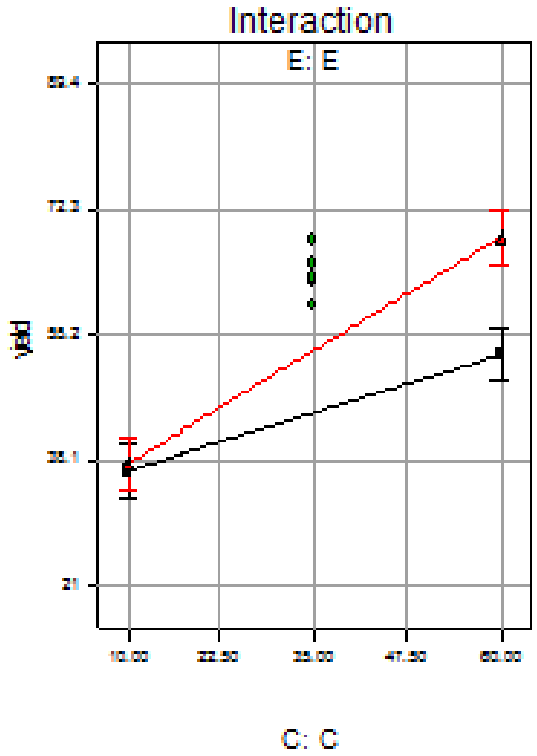
B= [NaOH]

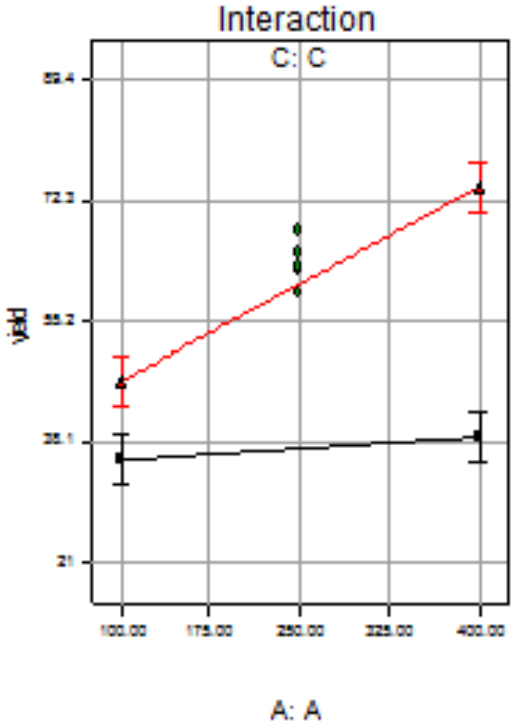
D=temperature

A=molar ratio

C= reaction time

E=mixing speed


Interaction
E: E
yield
C: C


Interaction
C: C
yield
A: A

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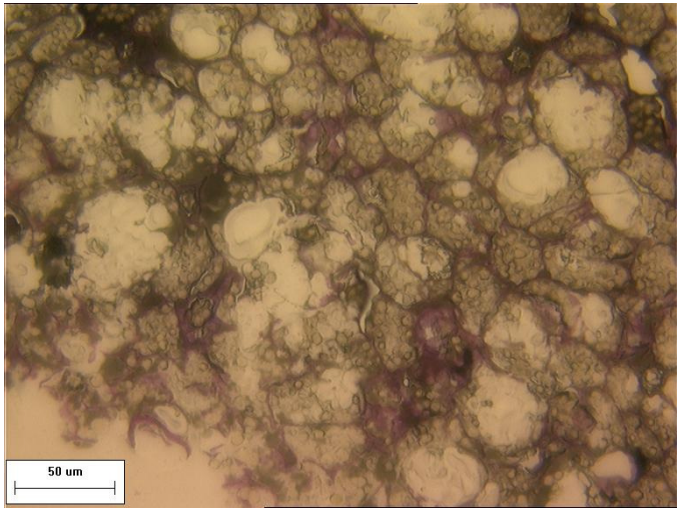

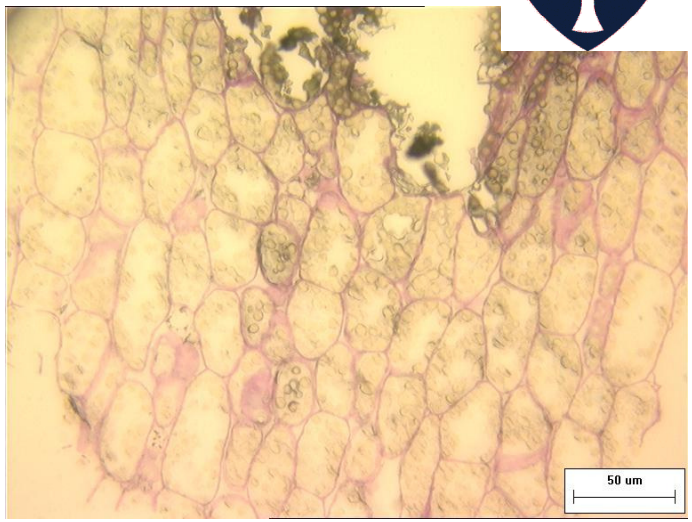

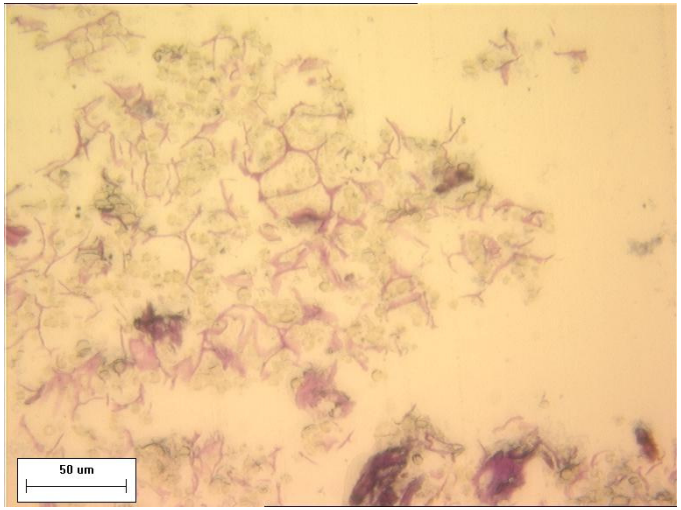

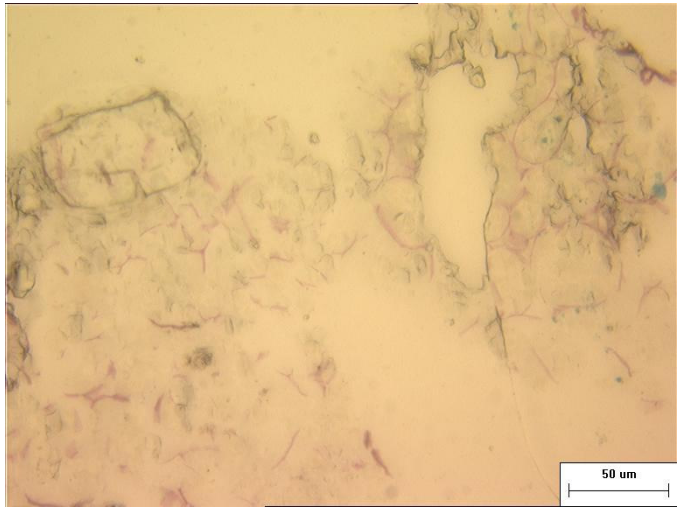

Clockwise from top: Before reaction, 100 molar, 300 molar, 500 molar

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QuickTime™ and a
TIFF (Uncompressed) decompressor
are needed to see this picture.

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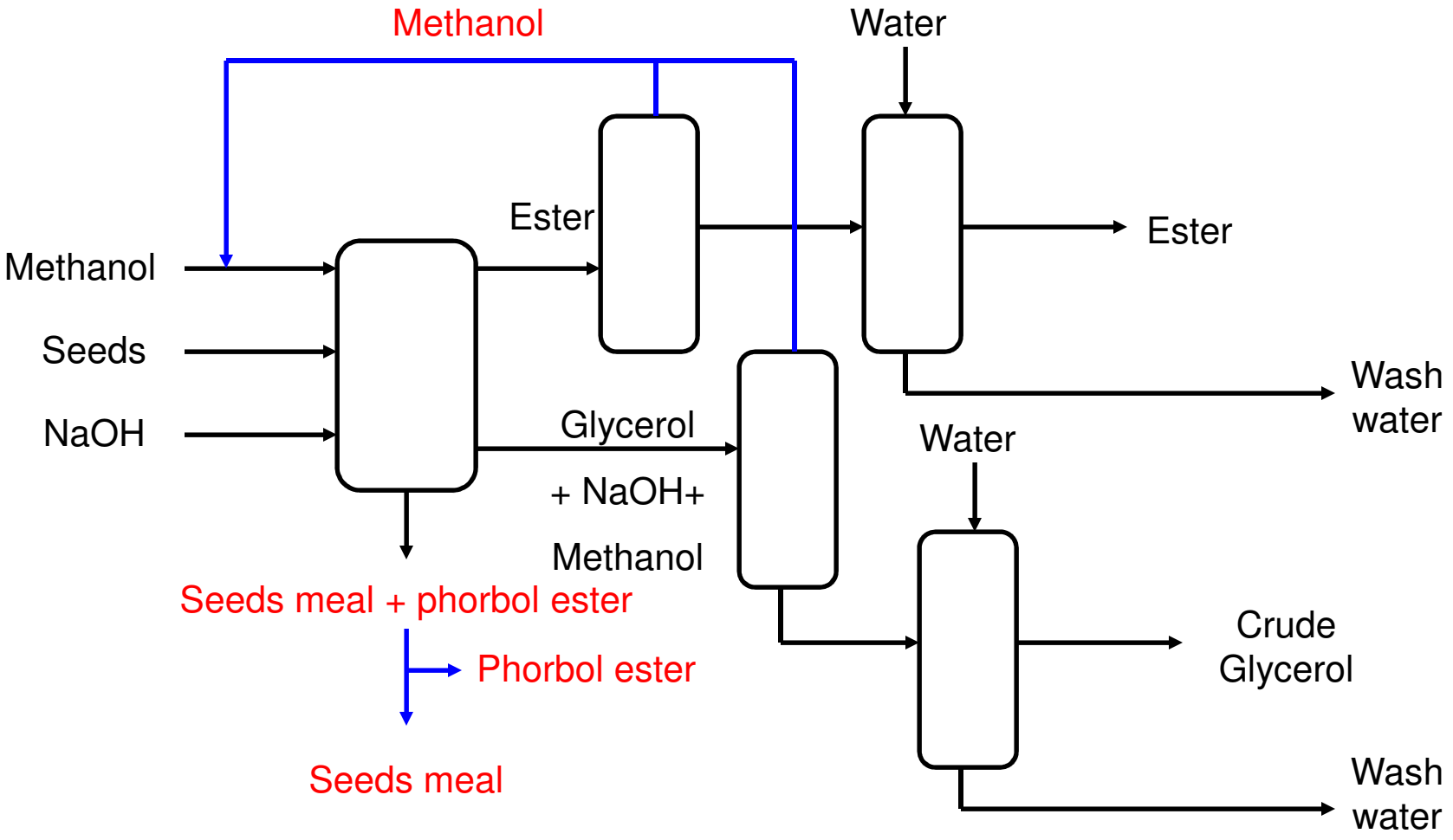
Before Reaction

After Reaction

Next: Biorefinery concepts?



• A (molar ratio), C (reaction time) and E (mixing speed) are significant to the biodiesel yield

• B (catalyst concentration) and D (reaction temperature) are not significant to the biodiesel yield

• Interaction between A/C and C/E also significant to the yield

• The presence of curvature also significant, indicating that first order model is not suitable to predict the model

• Higher order model is needed to predict the model more accurately

• Optimal condition within this parameter space are:

- 60 minute reaction time
- 400 rpm agitation speed
- 45^{o}C reaction temperature
- 0.17 N of NaOH concentration, and
- 400 molar ratio of methanol to oil

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